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ORIGINAL ARTICLE

# Eco-friendly and protective natural dye from red prickly pear (*Opuntia Lasiacantha* Pfeiffer) plant

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**Abstract** New natural dye extracted from red prickly pear was used for dyeing wool with different types of mordents. The effect of mordant concentration on the color strength was discussed; the results obtained indicated that the color strength decreases with the increase of mordant concentration. The effect of the dye bath pH, salt concentration, dyeing temperature and dyeing time was also studied. The color strength and the dye uptake have exhibited high values. Good fastness properties of the dyed fabric were achieved.

Antimicrobial activity of wool fabric dyed with this dye was tested according to diffusion agented. Test organisms *as Escherichia coli, Bacillus subitilus, Pseudomons aeruginosa* and *Staphylococcus aureus* were used and the results indicated that the samples exhibited a high inhibition zone.

According to the available literature, this is the first report concerning a natural dye for fabric from fruits of red prickly pear plants.



## 1. Introduction

Red prickly pear (*Opuntia Lasiacantha* Pfeiffer) is one of the plants of cactacoae family. The plant grows in many parts of the world such as Africa, Australia and the Mediterranean basin. In recent years according to field observations the area of prickly pear cultivation was increased rapidly with reclamation of new lands in the desert in Egypt. The fruits of the plant (Fig. 1) are used for many foods and medical industries.

Prickly pear juice is used as a natural dye. Betalain pigment was isolated from prickly pear (Fernandez-Lopez and Almela, 2001; Forni et al., 1992), it is found also in red beets (*Beta vulgaris*). The betalains are a group of nitrogen containing pigments that are yellow, orange, pink, red and purple in color. Betalains have no toxic effects in the human body and are seen as a natural and safe alternative to synthetic red coloring (Pigi et al., 2003; Ramadan and Morsel, 2003a,b). In food industry, there is a growing tendency to replace synthetic dyes by natural pigments as red prickly pear (Cai and Corke, 2000; Saenz, 2002). Betalains are cationized compounds with

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**Figure 1** Red prickly pear fruit.

positive nitrogen in polyene system. 5-O –glucose betanidine (**1**) are the pigments of the cactus pear fruit. The main fox of interest, however, has recently been on betalain pigments as antioxidants (Stintzing et al., 2001; Stintzing et al., 2003). Betalains stability is affected by temperature, pH, oxygen, light, and aqueous activity. The use of prickly pears as a source of betalains may be interesting comparing with red beets since the plants of the Opuntia genus need mineral requirements from soil and water. This way may be a great alternative to agricultural economy in arid and semiarid regions (Castellar et al., 2003).

In the present paper betalain pigment extracted from red prickly pear juice was used with different mordents as natural dye for dyeing wool.

## 2. Experimental

### *2.1. Fruits of red prickly pear plant*

The fruits of red prickly pear plant were collected from different locations of Nobaria region (Beheria governorate at North West of Delta, Egypt) .The juice of mature fruits was used as natural source of dyes and anti fungal, anti bacterial agents against some microorganisms in other studies.

### *2.2. Extraction*

The peeled fruits of red prickly pear were homogenized with an equal amount of water. The mixture was heated for 5 min at 80 °C and quickly cooled on an ice bath until it reached a temperature of 8–10 °C then the extract was centrifuged for 20 min (Butera et al., 2002).

### *2.3. Materials*

Betalain pigment extracted from red prickly pear was used as a natural dye; the chemical structure of this pigment is derived from betalamic acid on the united components to this structure (Fig. 2).

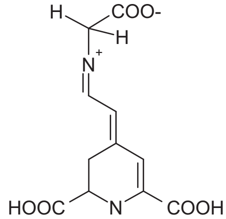

**Figure 2** Betalain pigment structure.

Scoured and bleached wool fabric with the following characteristics was purchased from Misr for Spinning and Weaving Company, Mahalla El-Kobra, Egypt; weight 205 g $m^{-2}$, 72 ends per inch, 64 picks per inch. Before using, the fabric was treated with a solution containing 5 g $L^{-1}$ non-ionic detergent (Hostapal CV, Clariant), at 50 °C for 30 min.

Then, the fabric was thoroughly washed with water and air dried at room temperature.

### *2.4. Mordanting*

Pre and post mordanting were done by using different mordents, namely, ferrous sulfate, copper sulfate, potassium dichromate and tannic acid with concentrations varied between (20, 40, 60, 80, 100) g/kg at optimized extraction and dyeing conditions (Bechtold et al., 2003; Bechtold et al., 2006).

### *2.5. Dyeing procedure*

Wool samples were dyed using a dye bath containing different amounts of sodium chloride (0–20 g $L^{-1}$) and the calculated amount of the dye with liquor ratio 40:1, heating at different duration (12–120) min and different temperatures (30–100 °C).

The dyed samples were rinsed with cold water, washed in a bath at liquor ratio 40:1 containing 3 g/$L^{-1}$ non ionic detergent (Hostapal CV, Clariant) at 50 °C for 30 min, then rinsed and finally dried at ambient temperature.

The pH values were recorded with Hanna pH meter and adjusted with dilute solutions of sodium carbonate.

### *2.6. Color strength*

The reflectance of the soaped samples was measured on a Perkin–Elmer Lambda 3B UV/Vis spectrophotometer. Relative color strengths (K/S values) were determined using the Kubelka–Munk equation (Judd and Wysezchi, 1975).

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-R)^2}{2R_0} \tag{1}$$

where $R$ = decimal fraction of the reflactance of dyed fabric, $R_0$ = decimal fraction of the reflactance of undyed fabric, $K$ = absorption coeffictent, and $S$ = scattering coefficient.

### *2.7. Fastness testing*

The dyed samples were tested according to ISO standard methods. The specific tests were: ISO 105 X12(1987), color fastness to rubbing; ISO 105-C02(1989), color fastness to washing; ISO 105-E04(1989), color fastness to perspiration; and ISO 105-B02 (1988), color fastness to light (carbon arc) **(**Handbook of Textite testing, 1988**)**.

## 3. Results and discussion

The obtained results suggest that the colorant from red prickly pear can be considered a potential source of natural food coloring (Fernandez-Lopez and Almela, 2001; Kanner et al., 2001). In our study, betalain pigment extracted from red prickly pear is used for dyeing wool; this dye is water soluble and has to be fixed to make the color fast or permanent, using fixatives or mordents, and it is stable in pH range of 4–7.

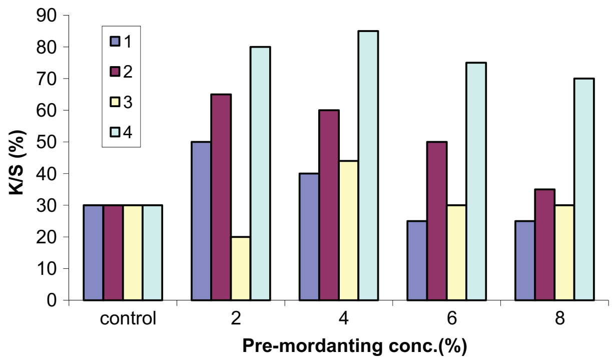


**Figure 3** The effect of the type of pre mordant on the color strength values at different concentrations of mordants. 1- Ferrous sulphate, 2- Potassium dichromate, 3- Copper sulphate, 4- Tannic acid.

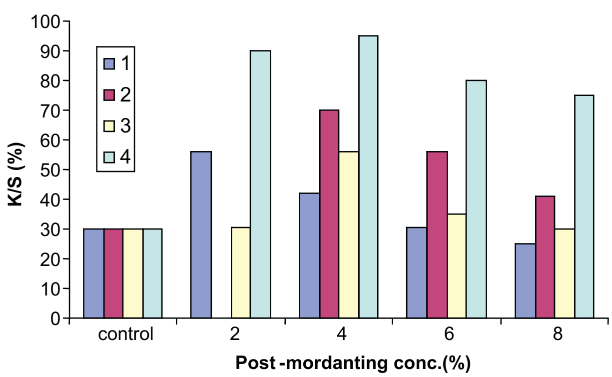


**Figure 4** The effect of the type of post mordant on the color strength Fig. 2: The effect of the type of pre mordant on the color difference values at different concentrations of mordants. 1-ferrous sulphate, 2-potassium dichromate, 3-copper sulphate, 4-tannic acid.

The stability of this dye was pH dependant and showed a maximum at pH 5 as shown in Fig. 3.

### 3.1. Dyeing

Reduction in the color strength with excess concentration of mordents (Figs. 3 and 4) was due to the aggregation of the extract molecules by the addition of excess metallic salts, causes a reduction in the extract solubility, which leads to its precipitation and difficulty of penetration during dyeing.

#### 3.1.1. Effect of mordents (type & conc.)

The colors produced from using different types of mordents are illustrated in Table 1.

#### 3.1.2. Effect of pre and post mordenting

In the cases of pre and post mordenting, the $K/S$ values increases with an increase in the percentage of the mordant until 4% concentration is achieved as shown in Figs. 3 and 4, By using a 4% solution of mordant, the results are even. After that unevenness, it is observed that there are no differences in the shade of the samples dyed with mordant solution and those dyed without a mordant solution. It is clear that the $K/S$ values decrease as the concentration of the mordant increases. It might be possible that the complex formed between fabric and mordant is too strong. The affinity formed between fabrics and mordant was strong .The affinity between the fabric and mordant increased as we increased the concentration, so that a stable complex was formed. Then when this mordented fabric was treated with dye extract, the dye was more rushed towards the fabric and caused unevenness. From the Figs. 3 and 4) it is clear that maximum $K/S$ is obtained for samples dyed with a 4% mordant concentration and then after that unevenness appears but the shade remains approximately the same.

The reduction in the $K/S$ with an excess concentration of mordents is due to the aggregation of the extract molecules by the addition of excess metallic salts, which causes a reduction in the extract solubility, and leads to its precipitation and difficulty of penetration during dyeing.

Effect of pre and post mordenting was also investigated for dyeing at optimum conditions. It is found that post-mordenting gives maximum colour strength as compared to pre-mordenting. The colour strength values are given in Figs. 3 and 4. Low colour strength in pre-mordenting condition is due to accumulation of the metal dye complex in the form of clusters, which upon investigation in spectra flash spectrophotometer show dull red and blue shades as shown in Table 2.

The above mordents used had the affinity for both the coloring matter and the fiber. The COOH groups present in the extract structure might affect greatly its sensitivity to the addition of metallic salts, and the efficiency of metal complex formation.

#### 3.1.3. Effect of dye bath pH

Fig. 5 shows that the pH values of the dye bath have a considerable effect on the dye ability of wool fabrics using the prickly pear dye. The effect of the dye bath pH can be attributed to the

**Table 1** Colors produced from using different types of mordents.

| Type of mordents | The produced color |
|---|---|
| Without mordant | Yellow |
| Ferrous sulphate | Yellowish brown |
| Copper sulphate | Green |
| Potassium dichromate | Olive green |
| Tannic acid | Red |

**Table 2** Diameter of inhibition zone inhibition (mm) in the diffusion agar test for the wool dyed with Perkily pear dye against selected microbes.

| Dye conc. (%) | Diameter of inhibition zone (mm) | | | |
|---|---|---|---|---|
| | Tested microbes | | | |
| | *E. coli* | *B. sub.* | *Ps. Au* | *Sta. A.* |
| 0.5 | 7.54 | 22.3 | 8.5 | 7 |
| 1.0 | 12.6 | 21 | 13 | 8.7 |
| 2 | 13.5 | 27.9 | 18.32 | 15 |
| 0.0 | 6 | 8 | 4 | 0 |

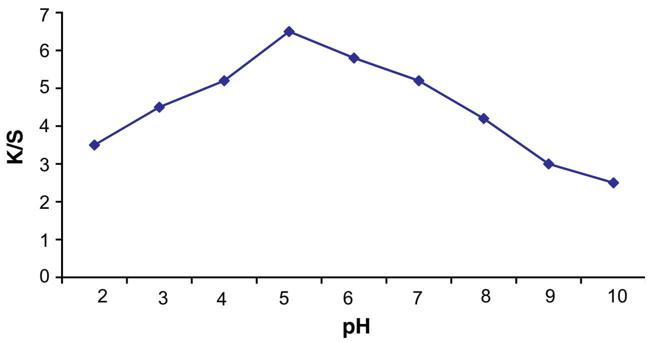


**Figure 5** Effect of dye bath pH on the color strength on wool fabrics. Dyeing conditions: 2% shade, L.R 40:1, at 100 °C, 40 min for 1 h.

correlation between dye structure and wool fabric. Since the dye used is sparingly soluble in water, containing COOH groups (Slawomir and Mizrahi, 2002; Castellar et al., 2003), thus it would interact ionically with the protonated terminal amino groups of wool fibers at acidic pH via ion exchange reaction due to the acidic character of the COOH groups. The anion of the dye has complex characters, and when it is bound on the fiber, with ionic forces, this ionic attraction would increase the dye ability of the fiber as clearly observed in Fig. 5.

It was noticed from the figure that higher dye ability at pH 5, then the dye ability decreases due to decreasing the number of protonated terminal amino groups of wool fibers, therefore the ionic interaction decreases.

### 3.1.4. Effect of salt addition

Fig. 6 shows the effect of salt concentration on the color strength obtained for the dyed fabrics. It is clearly indicated that as the salt concentration increases the color strength decreases, it is also noticed that at zero concentration the value of the color strength was maximum, i.e. dyeing without salt addition is the best condition.

### 3.1.5. Effect of temperature

The effect of temperature on the dye ability of wool fabrics with prickly pear dye was studied at different temperatures (30–100 °C). As shown in Fig. 7, it is clear that the color strength increases with the increasing of dyeing temperature and reaches to maximum value at 100 C.

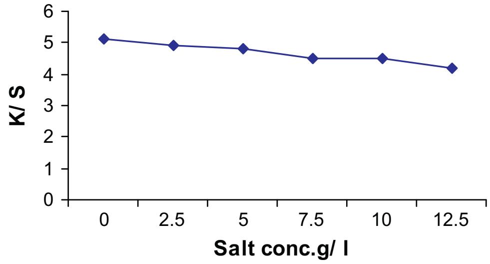


**Figure 6** Effect of salt addition to the dye bath on the colour strength of dyed wool. Dyeing conditions; 2% shade, L.R 40:1, 10 g $L^{-1}$ sodium chloride, 1 h, at 100 °C.

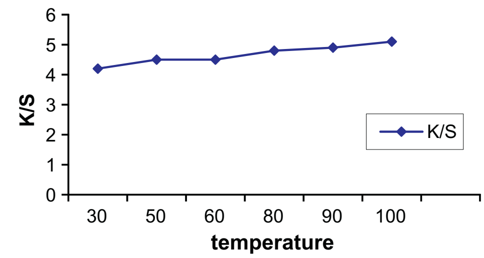


**Figure 7** Effect of the dyeing temperature on the color strength dyed wool fabrics. Dyeing conditions: 2% shade, L.R 40:1, pH 5, 1 h. 0 g salt conc.

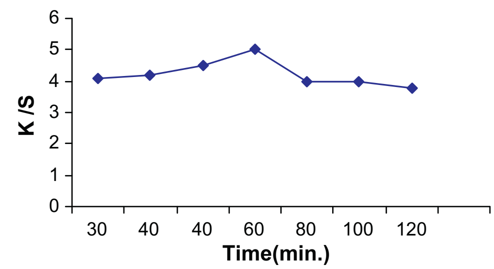


**Figure 8** Effect of dyeing time on the color strength of dyed wool fabrics. Dyeing conditions: 2% shade, L.R 40:1, pH 5 at 100 °C, 0 g salt conc.

### 3.1.6. Effect of dyeing time

As shown in Fig. 8, the color strength obtained increases as the time increases up to 60 min, then it decreases, i.e, $K/S$ displays maximum in 60 min-dyeing time.

## 3.2. Antimicrobial activity

The antimicrobial activity of different concentrations of dye of dyeing wool was studied. The data in Table 2 clearly show that an increase in dye concentration leads to increased inhibition zone of all tested microorganisms for dyed wool in comparison to undyed wool. It may be concluded that a high inhibition zone is recorded with 2% concentration. This indicates that the prickly pear dye is highly effective antimicrobial against all tested microorganisms. *Bacillus subtilus* and *E. coli* are highly affected by the dye. Meanwhile, *Staphyl A* shows a moderate effect.

## 3.3. Fastness properties

Fastness properties of the dyed fabrics are shown in Table 3. The results indicate good fastness properties of the mordanted dyed samples.

There was a relation between the dye mordant nature and the dye removal under the influence of washing solution and perspiration solution (acid and alkaline). This depends on the groups capable to form hydrogen bonding and metal complex. The higher the number of these groups the lower the magnitude of the dye removal, thus mordanting with tannic acid gave the highest washing fastness followed by potassium dichromate, copper sulphate, ferrous sulphate and the unmordanting samples; the same results were found for perspiration fastness.

**Table 3** Fastness properties of dyed wool fabrics.

| Types of mordents | Washing | | | Rubbing | | Perspiration | | | | | | Light fastness |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | a | | | b | | | |
| | A | C | W | Rd | Rw | A | C | W | A | C | W | |
| Without | 3 | 3–4 | 3–4 | 3–4 | 3 | 3–4 | 3 | 3–4 | 3–4 | 3 | 4 | 6 |
| Ferrous sulphate | 4 | 4 | 4 | 4–5 | 4–5 | 4 | 4 | 4 | 4 | 4 | 4 | 7 |
| Potassium dichromate | 4–5 | 4–5 | 4–5 | 5 | 5 | 5 | 4–5 | 5 | 4 | 4–5 | 5 | 7–8 |
| Copper sulphate | 4–5 | 4–5 | 4–5 | 4 | 4 | 4–5 | 4–5 | 4–5 | 4 | 4–5 | 4 | 7 |
| Tannic acid | 5 | 5 | 5 | 4-5 | 4-5 | 5 | 4–5 | 5 | 5 | 5 | 5 | 8 |

A = change in colour, C = staining on cotton, W = staining on wool, Rd = dry rubbing, Rw = wet rubbing, a = acidic, b = alkaline.

Rubbing causes the removal of adhered molecules which are most probably present as deposited layers on the fiber surface, while those forming intermolecular hydrogen bonding or being chemically combined with the fiber or forming metal complexation are not removed by rubbing, thus samples mordanted by potassium dichromate, tannic acid, ferrous sulphate and copper sulphate exhibited higher values of rubbing fastness than unmordanted samples.

The light fastness of colorants tends to fall when their particle size decreases, provided that no interfering factors operate. The effect is clearly due to the greater specific surface of colorants exposed to light; the increase of light fastness may be a result of the decreasing surface activity of the dye molecules and the increased dye–fiber bond strength, thus the light fastness of the dyed samples showed very good (7–8, 8) fastness with samples mordanted with potassium dichromate and tannic acid and good fastness (7, 7) with samples mordanted by copper sulphate and ferrous sulphate comparing with unmordanted samples.

## 4. Conclusion

New red pigment was extracted from prickly pear juice and used with different mordants as natural dye for dyeing wool with good fastness properties and high dye uptake.

Antimicrobial activity of wool fabric dyed with this dye was tested according to diffusion agented. Test organisms as *E. coli, B. subtilus Pseudomons aeruginosa* and *Staphylococcus aureus* were used and the results indicated that the samples exhibited a high inhibition zone. Our study shows that natural red prickly pear dye under investigation can provide bright hues and color fastness properties. They can serve as a noteworthy source of raw material in the future. Chemical modification of natural compounds such as the dye in question could be an interesting field of study as it could appreciably facilitate the synthesis of dye molecule.

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